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Report No. ARF-C6001-3
(Progress Report)

PREPARATION AND EVALUATION OF NEW HYDRAULIC FLUIDS

Bureau of Ships
Washington 25, D. C.

ARMOUR RESEARCH FOUNDATION OF ILLINOIS INSTITUTE OF TECHNOLOGY

AD 423950

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Progress Report

PREPARATION AND EVALUATION OF NEW HYDRAULIC FLUIDS

January 28 through February 27, 1963

Bureau of Ships
Washington 25, D. C.

Contract No. NObs-88249
ARF Project C6001

The purpose of this project is to develop new fire-resistant hydraulic fluids based on fluorinated, sulfur-containing compounds. The compounds will be synthesized specifically to meet the critical property requirements. Various derivatives of sulfur hexafluoride and other fluorinated materials are being investigated.

During the past month fluorination of di-n-propyl sulfide, $(C_3H_7)_2S$, in the Simon's cell was studied. In the first run, 7 ml of di-n-propyl sulfide was electrolyzed in approximately 50 ml of liquid hydrogen fluoride at -78° C. Electrolysis was carried out for approximately 12 hours at 3 to 5 volts (5 to 7 amps). After the run had been terminated, only a solid, powdery product was found in the cell. This product was not characterized because only fluorinated liquid products are of interest.

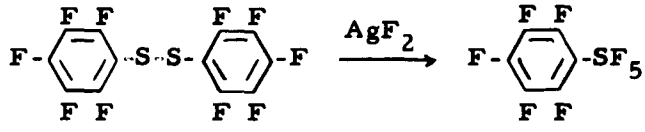
In the second run, 10 ml of di-n-propyl sulfide was electrolyzed in 40-50 ml* of liquid hydrogen fluoride. During this run, both the body of the cell and the reflux assembly were refrigerated with powdered dry ice. (In the first run only the reflux condenser had been refrigerated.) Electrolysis was

*It was difficult to estimate the exact amount of hydrogen fluoride used because it had been transferred from the conical bottom of the hydrogen fluoride reservoir.

carried out for approximately 14 hours at 2 volts (2.5 to 2.8 amps). After the run had been terminated, about 40 ml of a liquid material was drained from the cell. This fuming, red liquid is now being characterized.

Before the third run can be started, the hydrogen fluoride handling system must be repaired. At present, hydrogen fluoride gas escapes from the cylinder during transfer operations. Repair work is now in progress, and the third run will be carried out as soon as sufficient hydrogen fluoride has been transferred to the reservoir.

The preparation of pentafluorosulfur pentafluoride, $C_6F_5SF_5$, is being carried out as shown in Equation (1).



(1)

The preparation of the starting material was described in Report No. ARF-C6001-2. The disulfide and the silver difluoride were heated gradually to about 130°C. Reaction started at about 80°C and then subsided until the temperature reached about 130°C.

The nuclear magnetic resonance (NMR) spectrum of the product shows a complex mixture of compounds. The gas-liquid chromatography (GLC) analysis shows two main product peaks (See Figure 1), which represent roughly 95% of the sample. It was suspected that one of these peaks was due to pentafluorophenylsulfur trifluoride and that the other was due to the desired product, pentafluorosulfur pentafluoride.

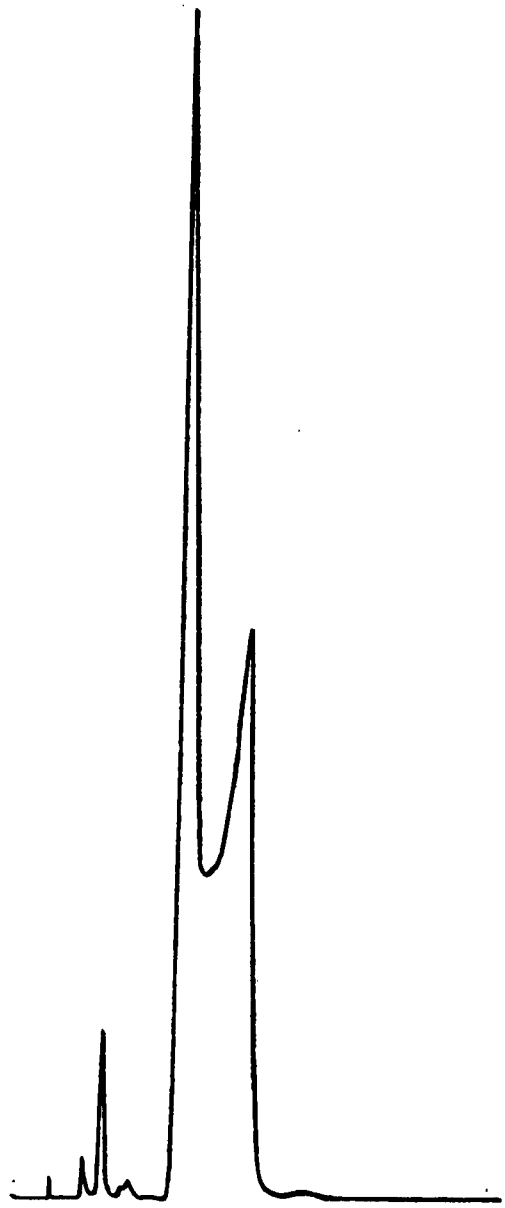


Figure 1

CHROMATOGRAPHIC ANALYSIS
OF PRODUCTS FROM REACTION 1

The trifluoride, $C_6F_5SF_3$, is easily hydrolyzed, whereas the pentafluoride, $C_6F_5SF_5$, should be stable to hydrolysis. Hence hydrolysis appeared to be a convenient way of disposing of the trifluoride and obtaining pure pentafluoride. Therefore, the sample was hydrolyzed, washed with sodium bicarbonate, and extracted with diethyl ether. The products from the hydrolysis should remain in the water extract. Chromatographic analysis of the product remaining in the ether extract showed one major peak, which differed from the product peaks shown in Figure 1. This new product peak falls between the other two on elution time. Upon closer examination of the peaks in Figure 1 it is seen that the product peak on the right side is unsymmetrical and appears to contain a third peak on the left shoulder.

It is now thought that this third product peak is due to the desired product and that the other two peaks may be due to C_6F_5SF and $C_6F_5SF_3$. The entire reaction and workup is now being repeated on a larger scale in order to obtain more of the product for anlysis.

In Report No. ARF-C60001-2, a large amount of data was given on the viscosities and other properties of various fluorocarbons. These data were gathered in order to determine the structural characteristics necessary for the desired viscosity index. A preliminary evaluation has shown the following:

(1) Straight chain compounds are better than branched compounds.

(2) Cyclic compounds are better than aliphatic compounds containing the same number of CF_x groups.

(3) Aromatic compounds are better than cyclic compounds.

During March the study of the preparation of $C_6F_5SF_5$ and $(C_6F_7)_2SF_4$ will continue. The synthesis of SF_5Cl will be started and should be completed by the end of March. This compound will be added to various olefins in order to prepare compounds of the type $Cl(CH_2)_xSF_5$ and $Cl(CF_2)_xSF_5$.

Data for this report are recorded in ARF Logbooks C 13206 and C 13209. In addition to the author, personnel contributing to the program include: T. Burgwald, Research Chemist; J. Raney, Associate Physicist; G Macur, Assistant Chemist; and R. Douthart, Assistant Chemist.

Respectfully submitted,

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IJS:lag
3-11-63

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